Electrical Resistance Change of Ceramic Matrix Composites in Response to Applied Load and Microstructural Damage

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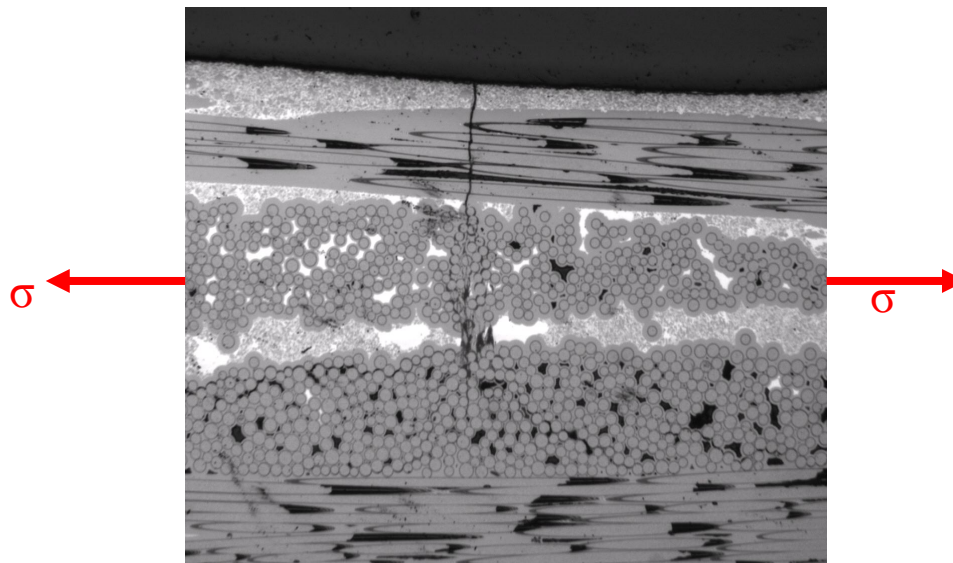
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The University of Akron

Why Electrical Resistance?

It would be beneficial to accurately detect small-scale transverse matrix cracks in a CMC coupon or component

As cracks form, only bridging fibers can carry current → resistance increases

For MI SiC/SiC, the matrix is more conductive than the fibers, which should give high sensitivity to crack formation

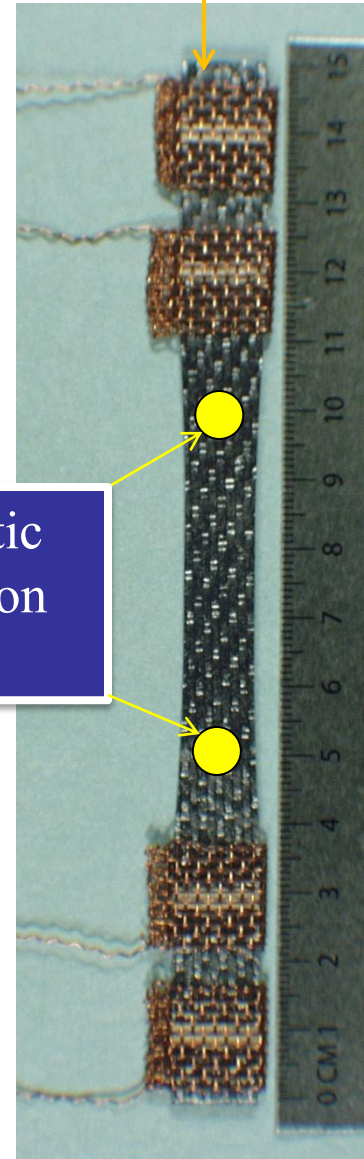**This is a relatively simple technique compared to other inspection methods**

- Resistance measured by four- point probe method
- Silver paint on surface for lower contact resistance
- Grip region wrapped with copper mesh (Used as electrical contact)
  - Offers a simple way of attaching electrical leads for elevated temperature tests
  - For select room-temperature samples, voltage of the contour gage section was also monitored
- Gripped with ceramic wedge grips (for electrical insulation)
- Capacitance strain gage used with 1% range over 25mm
- Resistance monitored with Agilent 34420A micro-Ohm meter
- Acoustic emission monitored by 50kHz to 2MHz sensors just outside the gage section

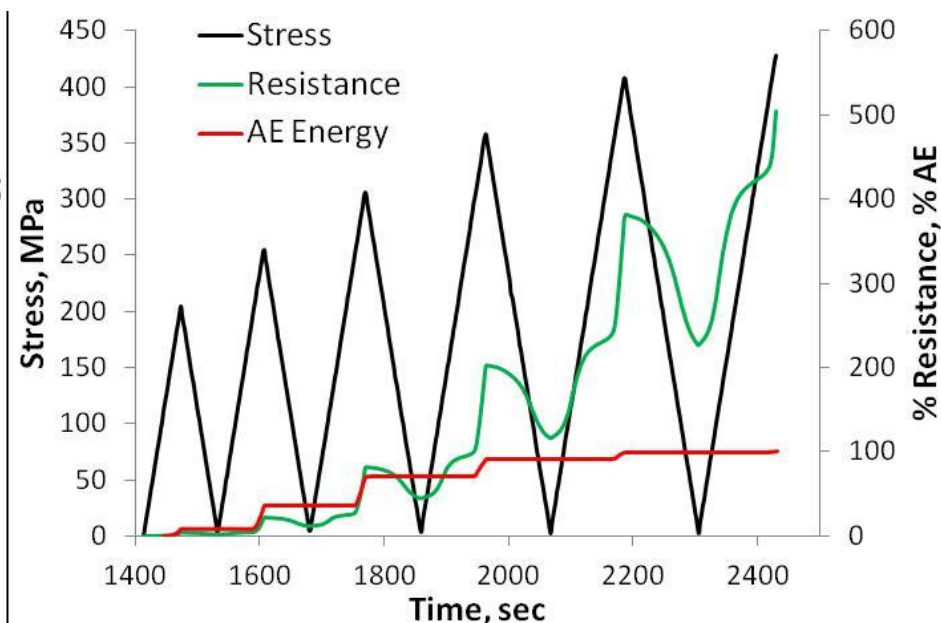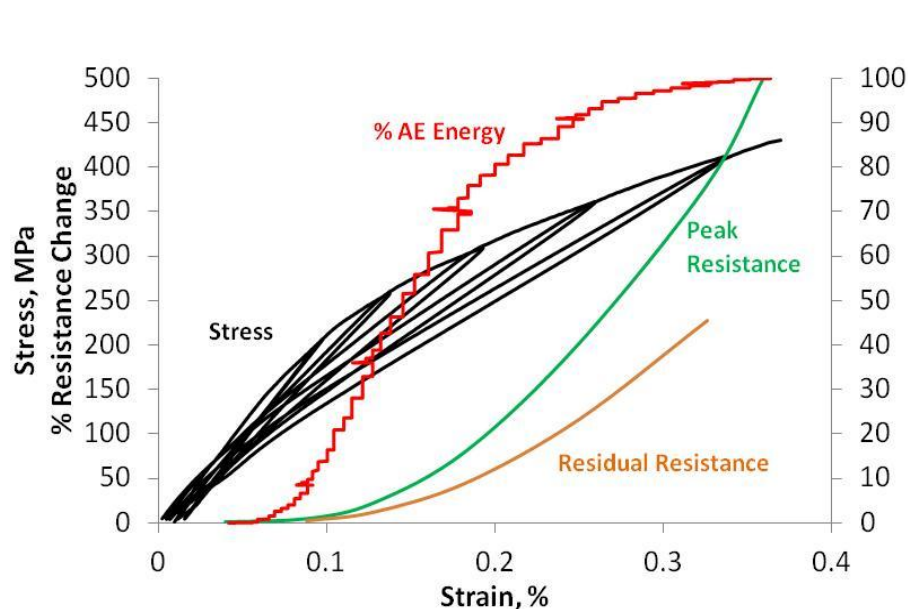Copper Mesh

Acoustic Emission (AE)

Syl-iBN/ Slurry Cast MI Matrix Woven Composite (f = 0.38)

Sample	Max Stress, MPa	Stiffness, GPa	Initial Resistivity, Ω-cm	Resistance change, %	Etched Crack Density, mm⁻¹	PL Stress, MPa	1st Loud AE Event, MPa	Stress where Resistance is non-linear, MPa
1	247*	246	0.027	17.9	0.9	170	138	140
2	300*	237	0.025	78.5	3	180	132	140
3	430	323	0.026	504	10	NA	103	115
4	440	253	0.023	580	--	180	139	110
5	442	260	0.024	466	11.5	170	147	150

Note: all samples have eight plies with a BN interphase, 800 fibers per tow, and total fiber volume fraction of 0.38

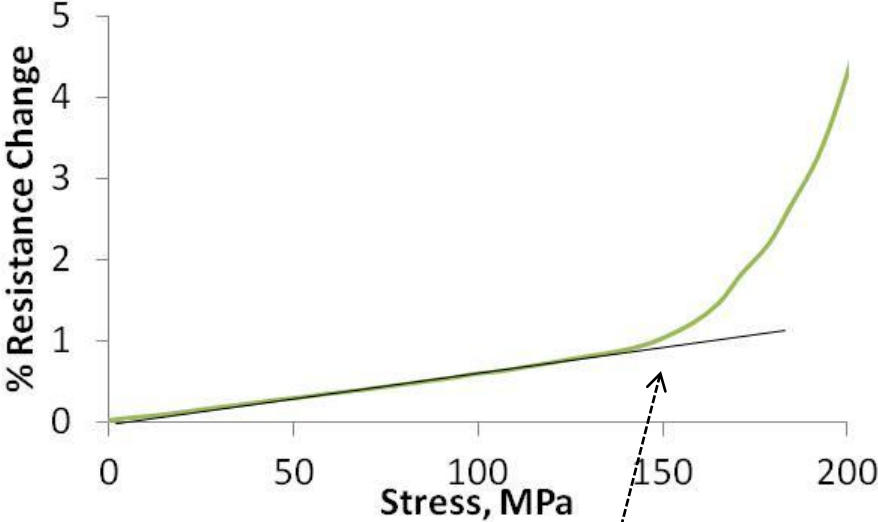* These samples were unloaded prior to failure to measure crack density

Syl-iBN/ Slurry Cast MI Matrix Woven Composite (f = 0.38)

Sample	M... St... M...
1	24...
2	30...
3	43...
4	44...
5	44...

PL Stress, MPa	1st Loud AE Event, MPa	Stress where Resistance is non-linear, MPa
170	138	140
180	132	140
NA	103	115
180	139	110
170	147	150

Note: all sam... w, and total fiber volume fraction of 0.38

* These sam...

GE Hi-NicS/ Pre-preg MI Matrix Laminate composite (f = 0.22)

Sample	Max Stress, MPa	Stiffness, GPa	Initial Resistivity, Ω-cm	Resistance change, %	PL Stress, MPa	1st Loud AE Event, MPa	Stress where Resistance is non-linear, MPa
0/90 balanced (0°)	274	288	0.024	118	248	208	230
0/90 balanced (+/-45°)	228	284	0.027	11.2	225	217	215
0/90 balanced (+20/-70°)	235	282	0.026	129	232	204	210
0/90 balanced (+70/-20°)	210	247	0.027	22.4	210	168	160
0/90 biased 2:1 in 0°	300	292	0.027	530	310	262	260

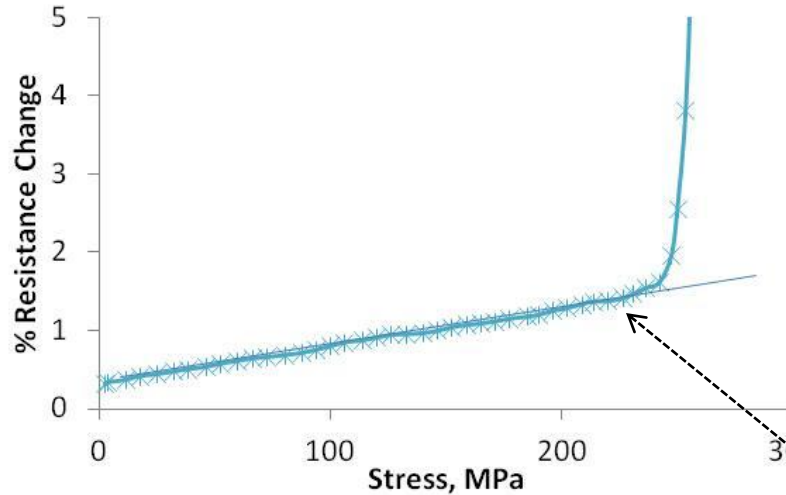Note: all samples have eight plies with a BN interphase and total fiber volume fraction of 0.22

GE Hi-NicS/ Pre-preg MI Matrix Laminate composite (f = 0.22)

Sample			PL Stress, MPa	1st Loud AE Event, MPa	Stress where Resistance is non-linear, MPa
0/90 balanced (			248	208	230
0/90 balanced (-			225	217	215
0/90 balanced (-			232	204	210
0/90 balanced (-			210	168	160
0/90 biased 2:1			310	262	260

Note: all sample ... olume fraction of 0.22
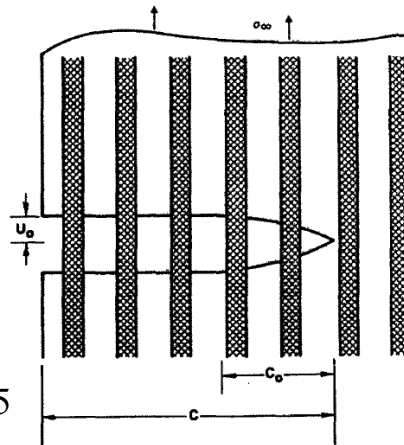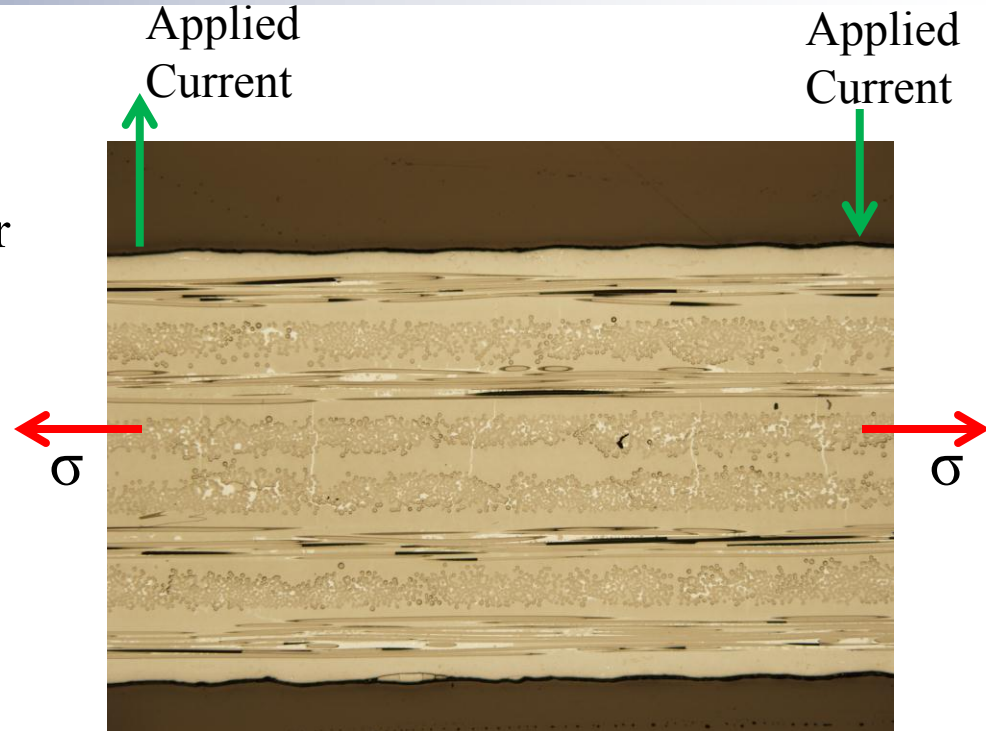

0/90 Balanced

Key Benefits

- Can be measured in-situ
- Resistance changes permanently for inspection at zero load
- The deviation from linearity correlates with the proportional limit stress
- Resistance is sensitive to crack formation
- Can be used at elevated temperature
- Repeatable

Key Concerns

- The data needs to be related to the microstructural changes in the material
- Many variables at room temperature
  - crack formation, crack growth, fiber/interphase sliding, fiber breaks
- Even more variables at elevated temp
  - creep of constituents, oxidation, change in resistivity with temperature
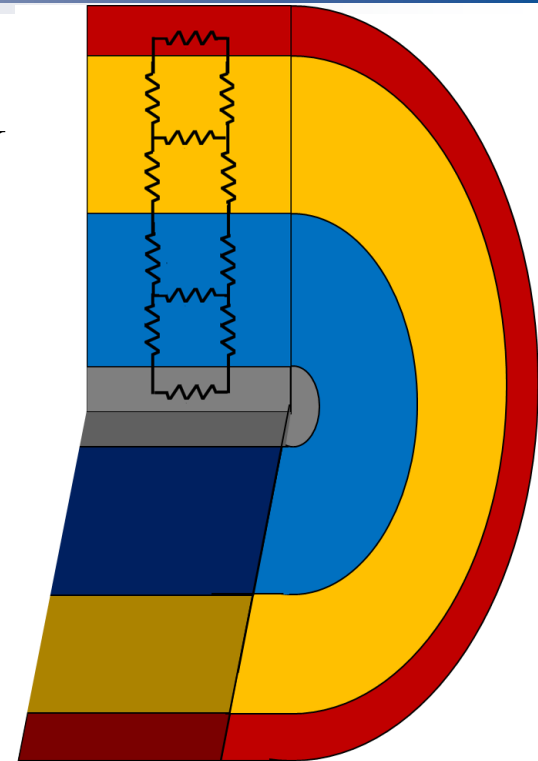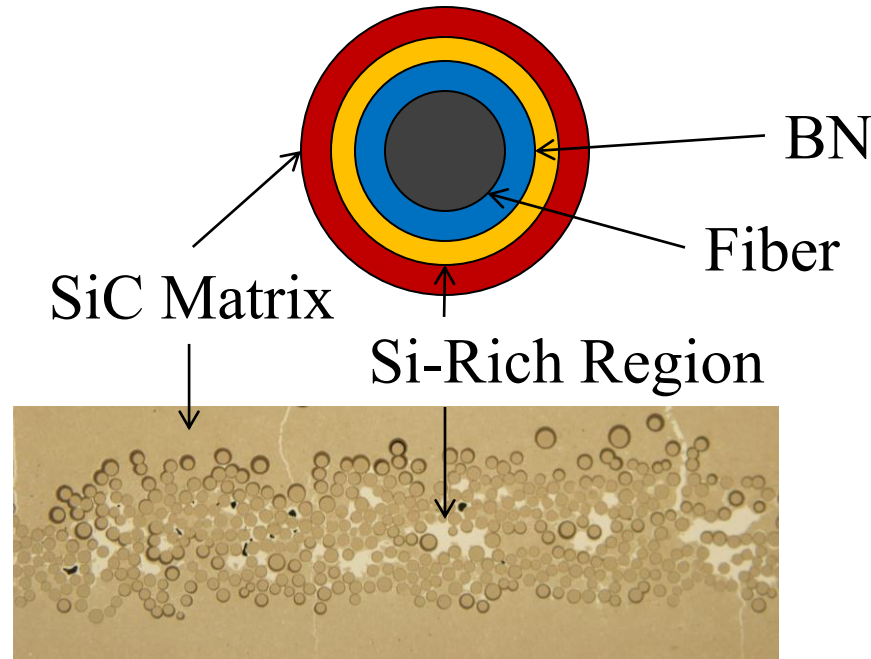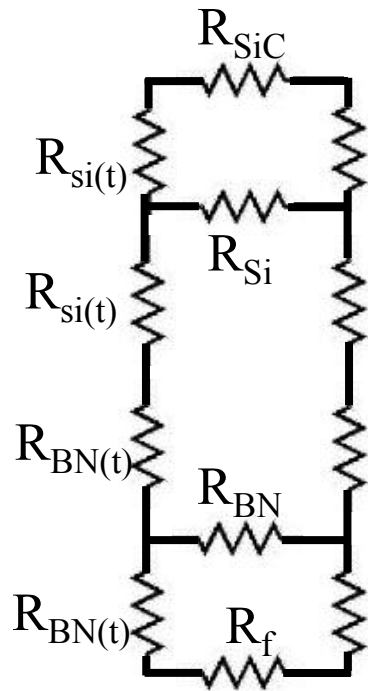- Need to understand what effect each mechanism has on resistance

- Current is applied at the MI matrix surface

- The matrix conductivity is an order of magnitude greater than the fibers (MI contains Si)

- Also, BN insulates the fibers from the matrix

- When the matrix is cracked, current must be transferred to the fibers

- The question is how?

Applied Current

Applied Current

σ

σ

Marshall, Cox, Evans 1985

- The composite is treated as a series of concentric cylinders
- Fibers are surrounded by BN, then a Si rich region in the ply, then the bulk MI
- All the fibers are treated as one, while maintaining the same relative volume fractions for constituents
- 90° plies are neglected in this case, since the fibers will not bridge the cracks and they do not provide a continuous path (BN interphases in series)
- Each unit cell represents a specified length along the loading direction (10 μm)

- Resistance along the length of each constituent:

$$R_c = \frac{\rho A_c}{L}$$

ρ is the constituent resistivity
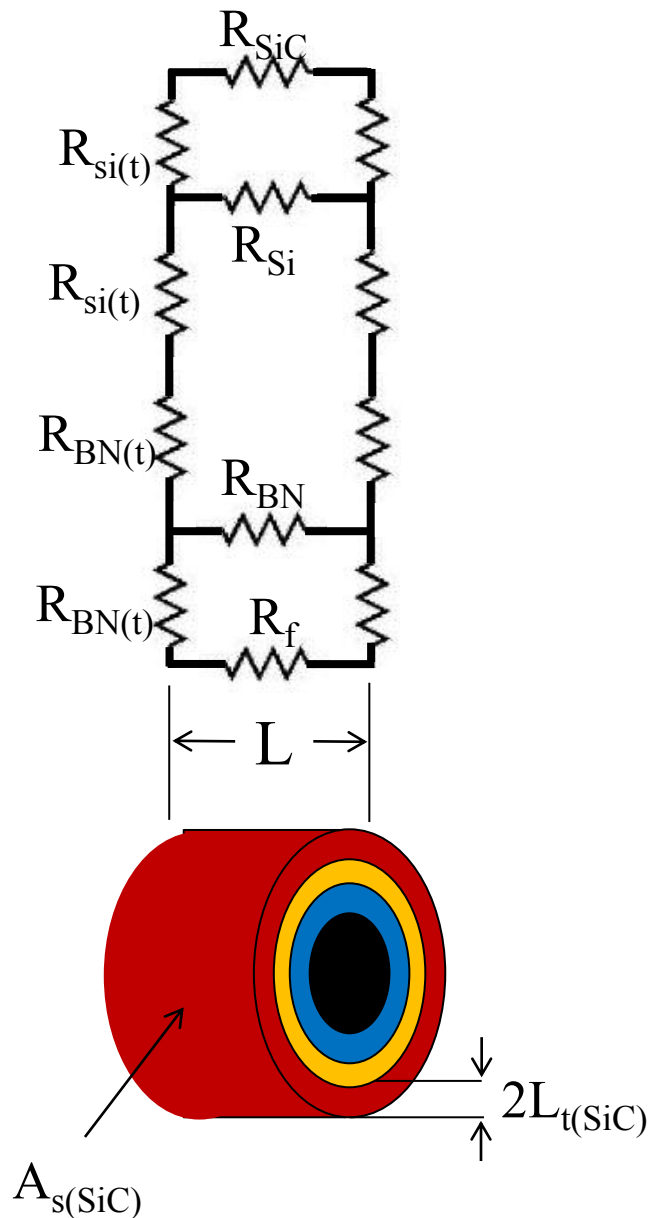A_c is the cross-sectional area
L is the length of the unit cell

- Similar equations describe the resistance of the constituents in the transverse (radial) direction:

$$R_t = \frac{\rho A_s}{L}$$

A_s is ½ of the mean surface area of the cylinder
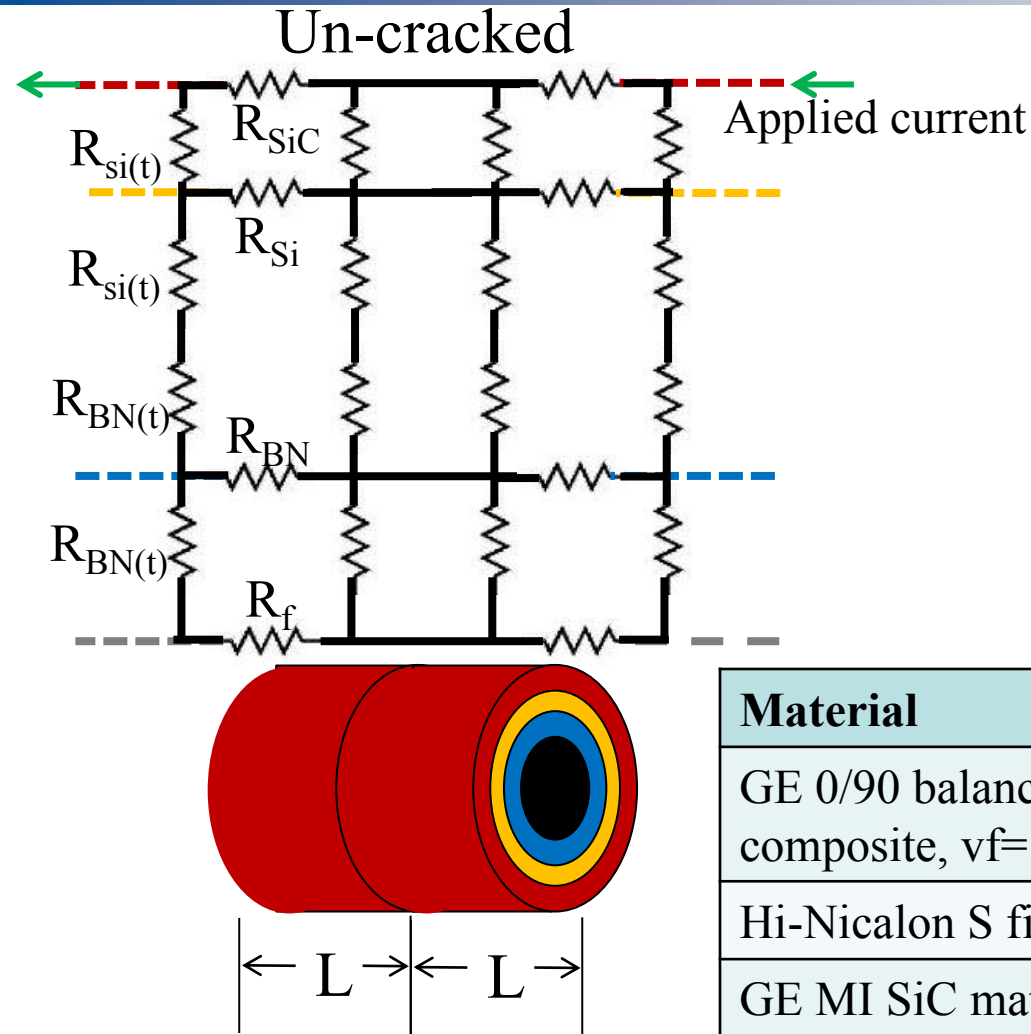L_t is ½ of the thickness of the cylinder

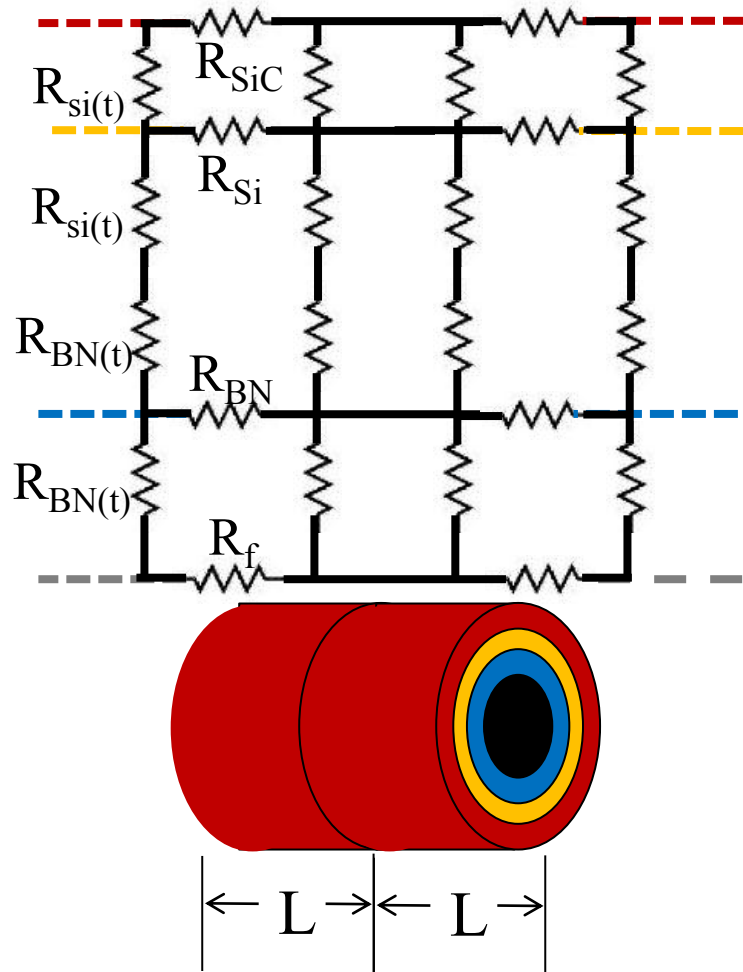| Material	Resistivity, Ω-cm
GE 0/90 balanced composite, vf= 0.22	0.027 (measured directly)
Hi-Nicalon S fiber	0.1 (from literature)
GE MI SiC matrix	0.037 (measured directly)
Si rich region in ply	Initially Unknown- very low
BN interphase	Initially Unknown- very high

Un-cracked

- Current is applied to the first and last cells at the outer SiC layer
- Adjusting the BN resistivity has no effect on the un-cracked composite since the matrix resistivity is low
- Resistivity of the Si-rich region can be adjusted to fit the initial composite resistivity

Material	Resistivity, Ω-cm
GE 0/90 balanced composite, vf= 0.22	0.027 (measured directly)
Hi-Nicalon S fiber	0.1 (from literature)
GE MI SiC matrix	0.037 (measured directly)
Si rich region in ply	0.0038 (from model)
BN interphase	Initially Unknown- very high

Un-cracked

R_{SiC}

$R_{si(t)}$

R_{Si}

$R_{si(t)}$

$R_{BN(t)}$

R_{BN}

$R_{BN(t)}$

R_f

L   L

Cracked

R_i R_i R_i R_i

R_{fu}

L   u   L

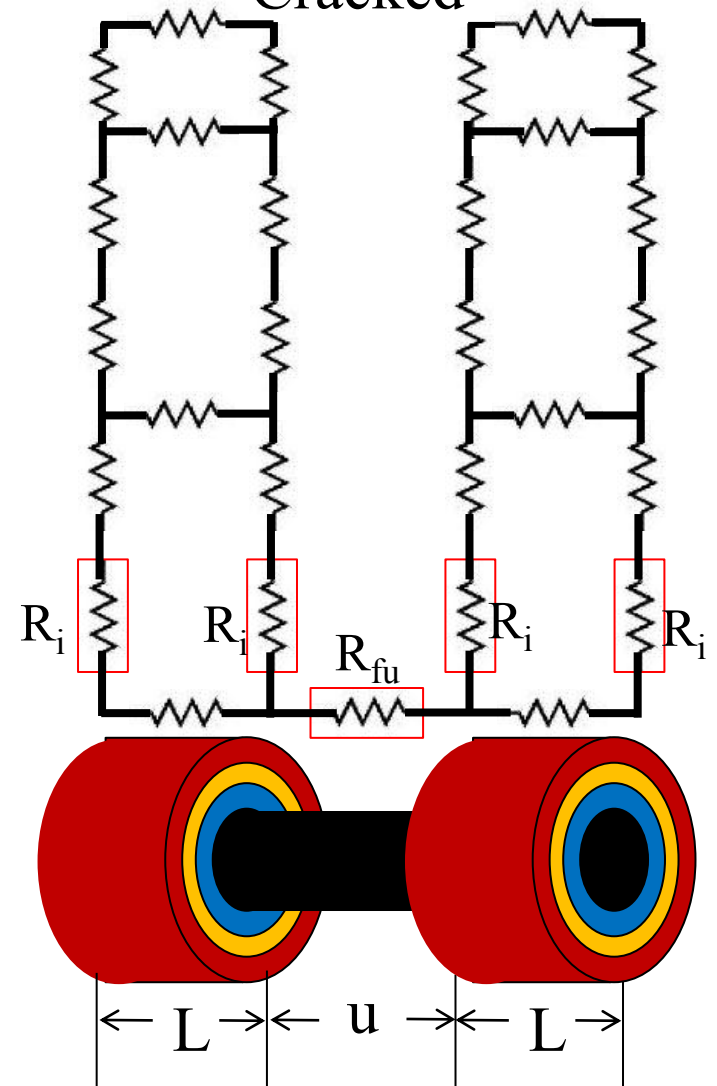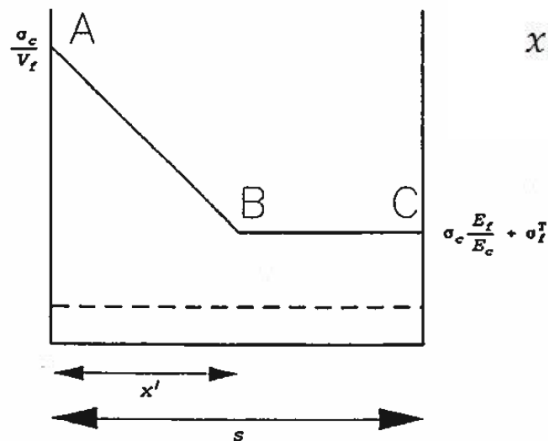# Model Description

Cracked

- The matrix and BN circuits are broken in the crack
- The fiber will de-bond from the BN and slide over a distance x^1
- An interfacial resistance is introduced between the BN and fiber
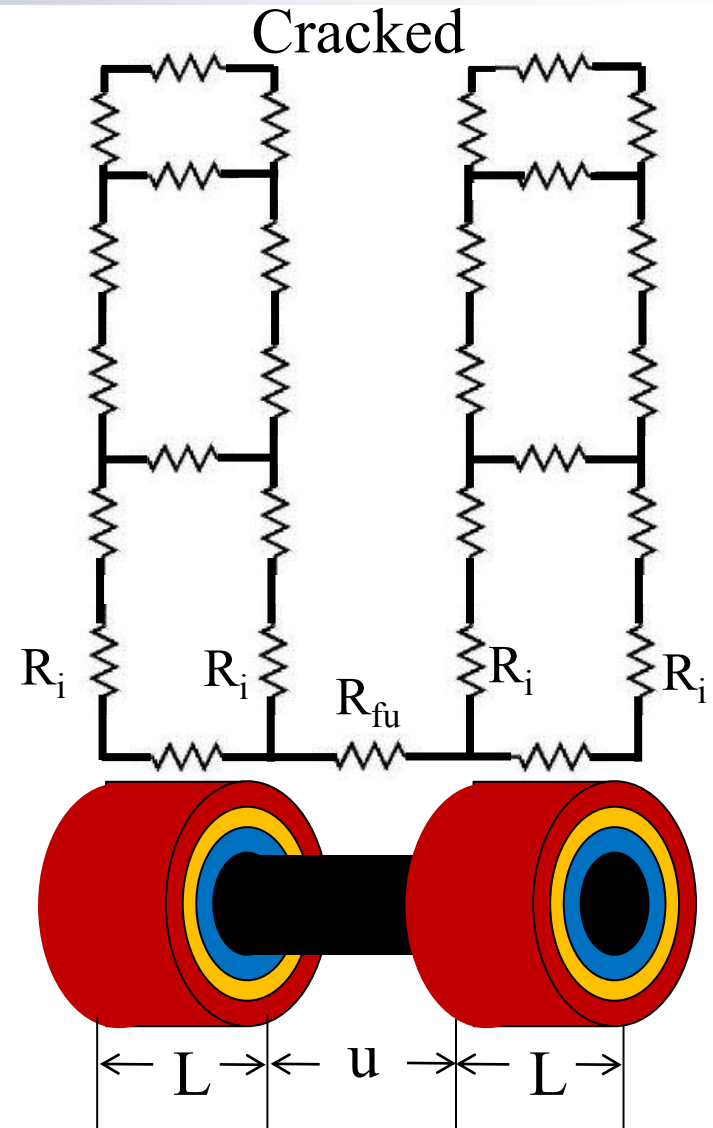- The surrounding unit cells within the distance x^1 will all be affected

$$x^1 = \frac{r}{2\tau}\left[\sigma_c \frac{v_m E_m}{v_f E_c} - \sigma_f^R\right]$$

Pryce, Smith 1993

8 Stress profile in the fibres for a cracked laminate at an applied stress σ_c

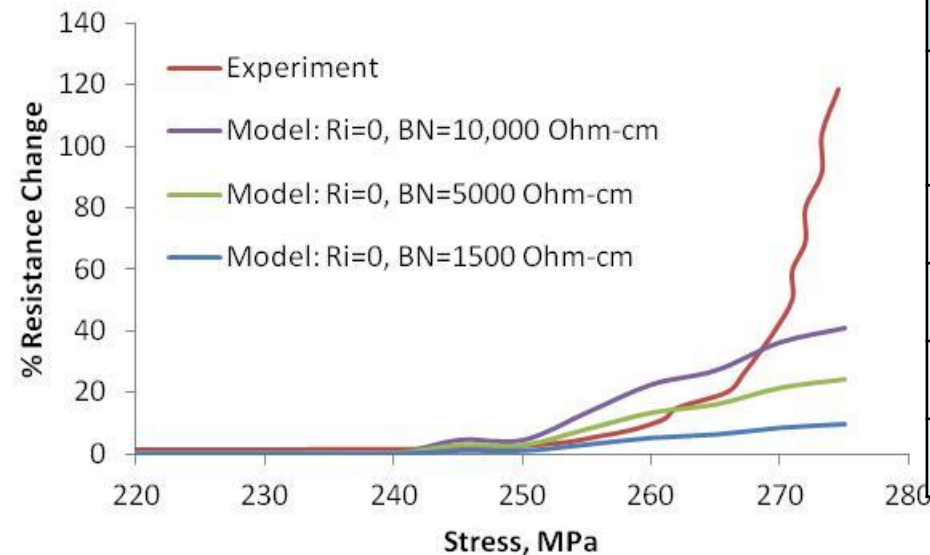$$u = \frac{\sigma^2 R}{4\tau v_f^2 E_f \left(1 + \frac{E_f v_f}{E_m (1 - v_f)}\right)}$$

Marshall, Cox, Evans 1985

$R_i \quad R_i \quad R_{fu} \quad R_i \quad R_i$

$\leftarrow L \rightarrow \leftarrow u \rightarrow \leftarrow L \rightarrow$

Model Calibration

- Acoustic emission energy is used as an estimate of crack density as a function of stress
- The model introduces cracks at incremental stress levels, according to the AE data
- The cracks are randomly distributed and the overlap length can be specified
- The resistivity of BN is determined by setting Ri=0 and adjusting the BN
- If the value is too high, the model will be too sensitive to cracks
- The maximum BN resistivity was chosen such that the model would never overshoot the experiment in the extreme case of Ri=0
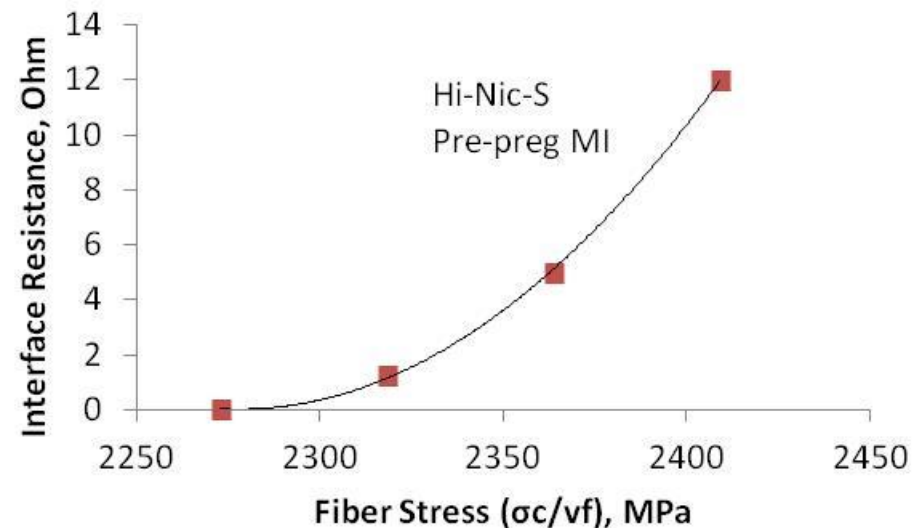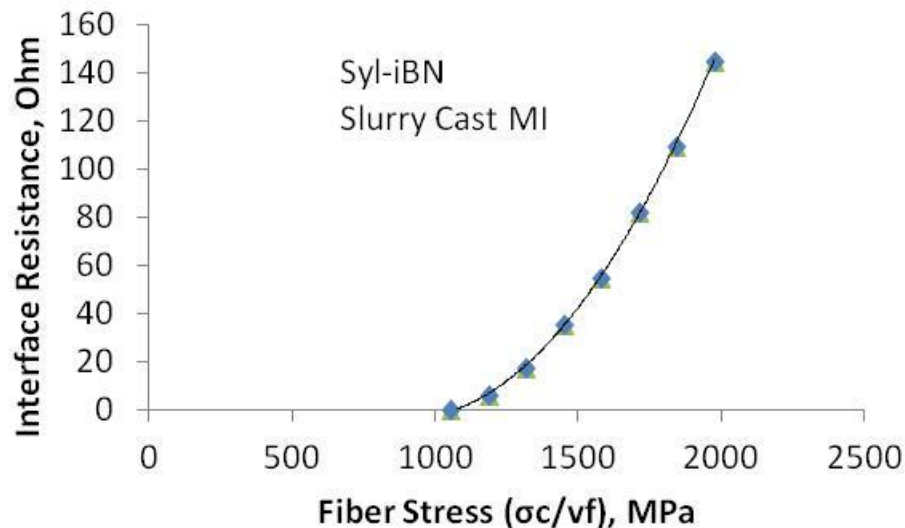

Material	Resistivity, Ω-cm
GE 0/90 balanced composite, vf= 0.22	0.027 (measured directly)
Hi-Nicalon S fiber	0.1 (from literature)
GE MI SiC matrix	0.037 (measured directly)
Si rich region in ply	0.0038 (from model)
BN interphase	1500 (from model)

- Interfacial resistance Ri is assumed to be uniform over the slip length x^1, but changing with stress
- It is expected that the interfacial resistance increases due to relative sliding between fiber and matrix
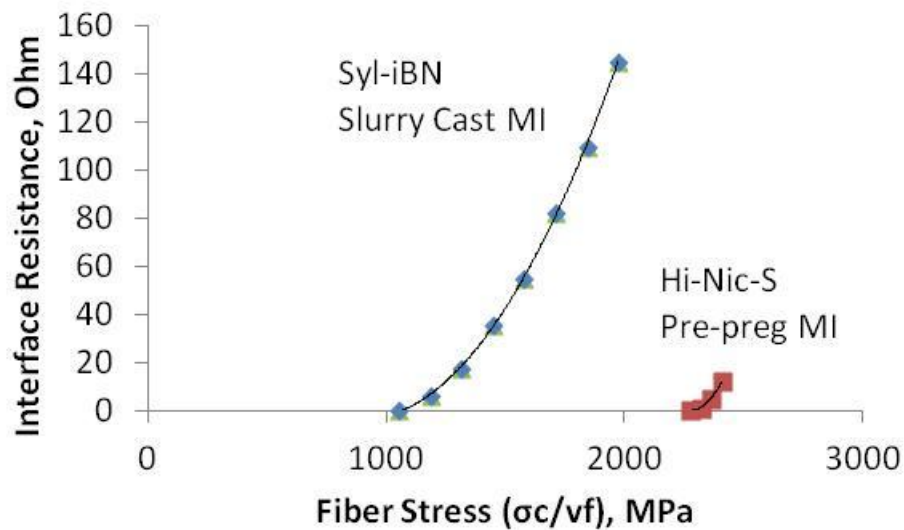- We see that Ri must be proportional to σ^2 to fit the experimental data

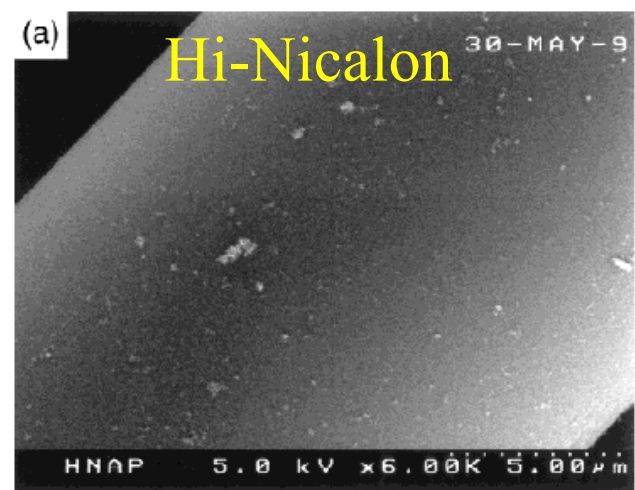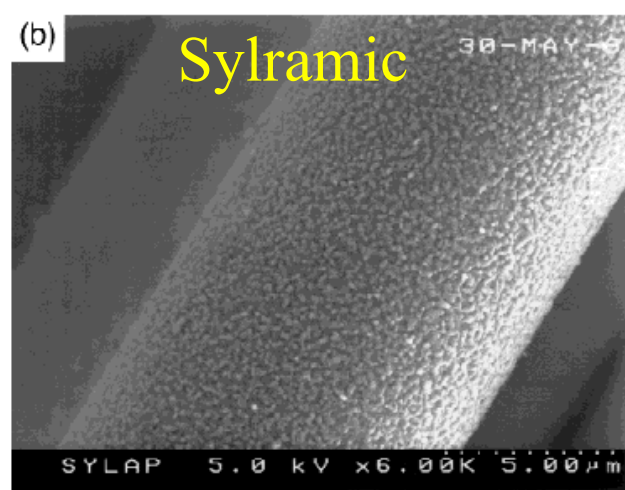- Sliding distance of the fiber at the crack surface is proportional to σ^2

$$u = \frac{\sigma^2 R}{4\tau v_f^2 E_f \left(1 + \frac{E_f v_f}{E_m (1 - v_f)} \right)}$$
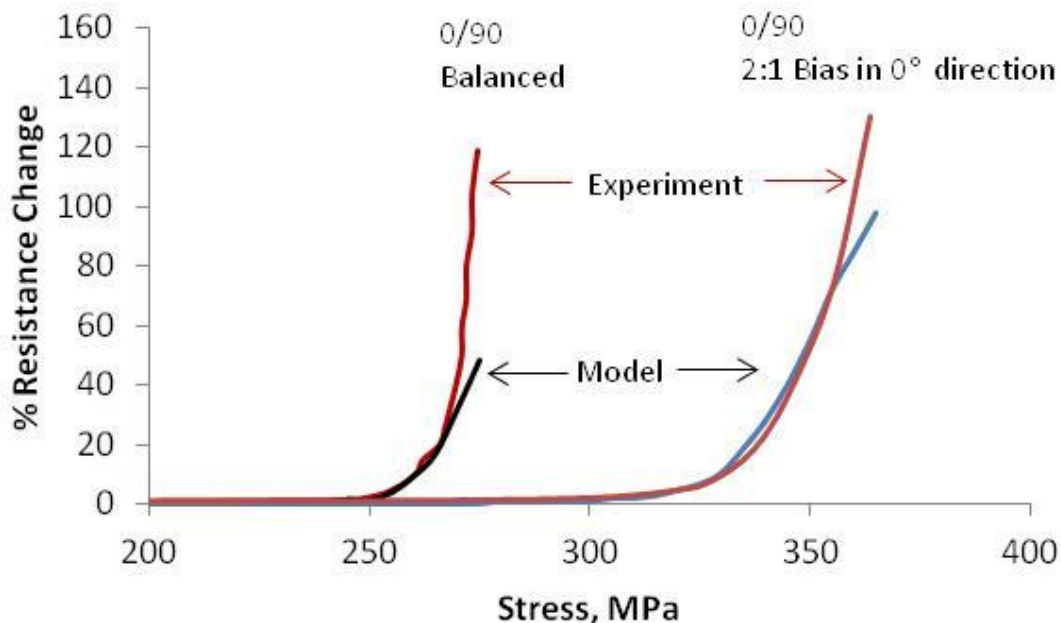
Marshall, Cox, Evans 1985

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- The difference in magnitude of Ri for the two composites is likely due to fiber roughness

Sylramic

Hi-Nicalon

- By using the parabolic relationship for Ri, the model fits the experiment for the balanced 0/90 GE pre-preg composite at 98% of the strength
- The same parabolic function generated from the balanced composite was used to model an unbalanced 0/90 sample with 2:1 bias in the loading direction
- The model also fit this data, which indicates that the relationship is more than a mere curve fit

- Electrical resistance offers a way of monitoring damage in CMC's

- Several factors influence the electrical properties

- A discrete model has been developed to understand the mechanisms causing electromechanical changes

- The model verifies that the interfacial resistance is a function of stress squared, consistent with fiber sliding

Examine the effect of the following:
- Ri varying along the slip length
- Fiber breaks
- Load-unload- reload
- Varying cross-section along the sample
- Temperature
- Creep
- Environment